

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

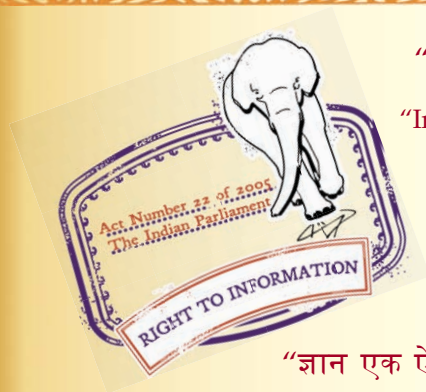
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 13072 (1991): Sulphur hexafluoride for electrical purposes [ETD 3: Fluids for Electrotechnical Applications]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



IS 13072 : 1991
IEC Pub 376 (1971)

Indian Standard

(Reaffirmed 2001)

SULPHUR HEXAFLUORIDE FOR ELECTRICAL PURPOSES — SPECIFICATION

UDC 621.315.618 : 661.21.487

© BIS 1991

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1991

Price Group 12

Indian Standard

SULPHUR HEXAFLUORIDE FOR ELECTRICAL PURPOSES — SPECIFICATION

NATIONAL FOREWORD

This Indian Standard, which is identical with IEC Pub 376 (1971) 'Specification and acceptance of new sulphur hexafluoride' issued by the International Electrotechnical Commission (IEC) was adopted by the Bureau of Indian Standards on the recommendation of the Fluids for Electrotechnical Applications Sectional Committee (ETD 03) and approval of the Electrotechnical Division Council.

The text of IEC standard has been approved as suitable for publication as Indian Standard without deviations. Certain conventions are however not identical to those used in Indian Standards. Attention is particularly drawn to the following:

Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

Text of Section 13 — Mineral oil content test, have been incorporated taking from First Supplement to IEC Pub 376 (1971).

Text of clause 26, dew point method, have been incorporated taking it from Second Supplement to IEC Pub 376 (1971).

Only the English language text in the International Standard has been retained while adopting it in this standard.

As in the Original Standard, this Page is Intentionally Left Blank

SECTION ONE — SCOPE

1. Scope

This Recommendation applies solely to new and unused sulphur hexafluoride, that is, as supplied by the chemical manufacturer and before introduction into any electrical equipment. It covers the properties and methods of test applicable to sulphur hexafluoride when this substance is supplied for use in connection with any electrical equipment.

SECTION TWO — GENERAL PROPERTIES OF SULPHUR HEXAFLUORIDE

2. Physical properties

Sulphur hexafluoride is a compound having the formula SF_6 . At normal room temperatures and pressures it is gaseous and has a density of 6.16 g/l at 20 °C and 760 torr (about five times the density of air). Since its critical temperature is 45.6 °C it can be liquefied by compression; it is normally transported as a liquid in cylinders. The saturated vapour pressure curve is shown in Figure 1.

The pure gas is colourless, it has no smell and it is non-toxic.

Warning. — Although the gas is non-toxic, it will not support life, and equipment containing sulphur hexafluoride must not be entered without adequate ventilation.

Similarly, since the gas has a much greater density than air, trenches and so forth may remain full of the gas and personnel must be made aware of the danger of asphyxiation in such places.

3. Electrical properties

The gas is electronegative (i.e., tends to attract free electrons) and has arc quenching properties and high electric strength. The electric strength is greatly affected by the conditions of test or of use, but for guidance, at about atmospheric pressure, under the usual conditions of test or use and in a uniform field, the electric strength may be expected to be about two and a half times that of nitrogen.

4. Compatibility

Pure sulphur hexafluoride is an inactive material. Up to temperatures of about 180 °C its compatibility with materials used in electrical construction is similar to that of nitrogen. Higher temperature operation may be possible with the pure gas but the merits of each case must be considered separately. Discharges in equipment cause dissociation of the pure gas; the breakdown products may be incompatible with constructional materials.

5. Effects of impurities

Any departure from the above properties is caused by impurities that are generally present in sulphur hexafluoride as supplied commercially; these impurities arise from the manufacturing processes, from leakage in manufacturing plant, or from contamination from the containing cylinders. Tests are carried out to detect these various impurities, and later sections of this Recommendation describe the nature and importance of such impurities, lay down limits for impurities so that new gas complying with this Recommendation is suitable for electrical applications, and specify methods to be used in testing for these impurities.

SECTION THREE — NATURE AND LIMITING QUANTITY OF IMPURITIES

6. Toxic impurities

Certain impurities that can be present in new gas are highly toxic, in concentrations so small that they cannot readily be determined by analytical methods. A biological method of test for toxic impurities is given in Clause 22; sulphur hexafluoride conforming to this Recommendation must comply with the requirements of that test.

Note. — It is important that users are assured that the gas conforms to the toxicity requirements. Attention is drawn to the provisions of Clause 15, which oblige the manufacturer to supply a certificate of non-toxicity if so requested.

7. Impurities affecting safety of equipment

Many categories of impurity must be limited to quantities such that either separately or in combination they will not present a hazard to the operation of the equipment in which the gas is to be used. For example, water, acidic impurities and oxygen (especially in combination) may promote corrosion leading to faulty mechanical operation; water in combination with any acidic impurities may condense at low operating temperatures and high operating pressures with danger to the electrical safety of the equipment. Thus the quantities of such impurities must be small so that corrosion or condensation is insignificant.

8. Impurities diluting the product

There may also be substances in the commercial gas which do not present any particular hazard to equipment, but are undesirable only because they are a dilution of a more valuable material, or because in certain circumstances they can lead to difficulties in subsequent handling. In practice, air is the only material likely to be found in substantial quantities. The air content and/or nitrogen and oxygen are determined together with carbon tetrafluoride by the method of Section Ten. Oxygen and nitrogen will not necessarily be present in the same proportions as in the atmosphere, due to the manufacturing processes.

Sulphur hexafluoride conforming to this Recommendation must not contain more than the maximum allowable quantities of impurities shown in Table 1.

TABLE I

Impurity or group of impurities	Maximum permitted concentration mass by mass	As determined by test method(s) of Section No.
CF ₄	0.05%	10
Oxygen + nitrogen, air	0.05%	10
Water	15 ppm	9
Acidity expressed as HF	0.3 ppm	12
Hydrolysable fluorides, expressed as HF	1.0 ppm	11
Oil content	See Note 3	13

Notes 1. — Industrial application of this gas is at present limited; the above provisional figures may be revised after more experience has been obtained.

2. — The purchaser may wish to specify a maximum condensation temperature in addition to the impurities listed in the table above. See Clause 23 for further details.

3. — SF₆ shall be substantially free from oil. The maximum permitted concentration of oil and the method of measurement are under consideration.

9. Impurities in relation to electric strength

Impurities have so little effect on the electric strength of sulphur hexafluoride that the impurities in the gas complying with the present Recommendation will have no detectable effect on the electric strength.

10. Impurities in relation to smell

Some impurities (mainly acidic impurities) that can be present in new gas have an odour, but the quantities of such impurities in gas conforming to this Recommendation are not large enough for any smell to be detectable by normal human beings.

Warning. — Gas must not be inhaled unless it is known to comply with the toxicity requirements.

SECTION FOUR — CONTAINERS FOR SULPHUR HEXAFLUORIDE

Preferred sizes of cylinders

The standard sizes of cylinders, expressed in litres, for SF₆ are: 3, 5, 10, 20, 40, 80, 150 and 500.

For preference 10 l and 40 l cylinders are to be used. The test pressure of the cylinders is to be 70 bars.

The maximum filling ratio of the cylinder must be in accordance with requirements in force in the country of use, transport regulations, etc.

Note. — In temperate countries a maximum filling ratio of 1.04 kg/l is usual. In tropical countries a smaller filling ratio (e.g. 0.75 kg/l) is often specified.

The mass of sulphur hexafluoride (kilogram) in each cylinder is to be stated with each cylinder.

12. Identification of cylinders

Each cylinder should be legibly marked at the valve end and preferably off the cylindrical part of the body with:

- a) the symbol SF_6 ;
- b) "sulphur hexafluoride" in words, in the language of the country of origin;
- c) the tare weight of the cylinder without protective cap.

Additional markings, colourings, etc., may be added in accordance with national or manufacturer's practice.

13. Cylinder valve outlets

This question is under consideration by ISO.

Pending the preparation and publication of an ISO Recommendation dealing with valve outlets for gas cylinders, ISO expressed the wish that a country still requiring a national standard valve outlet for sulphur hexafluoride cylinders should adopt the connection described in Figure 2.

SECTION FIVE — CONDUCT OF TESTS - GENERAL

14. Competence of the test laboratory

Many of the tests described necessitate specialized equipment, facilities, or skills (e.g., biological testing arrangements, gas chromatograph equipment, knowledge of high-sensitivity weighing techniques); it is essential that all tests are carried out by a properly competent organization using suitably experienced personnel.

15. Certification

If requested by a purchaser of the gas, the manufacturer shall supply a written statement or certificate that the new gas meets all the requirements (or by agreement, certain of the requirements) of this Recommendation.

SECTION SIX -- SAMPLING TECHNIQUE

16. General

Sulphur hexafluoride is normally supplied under pressure in cylinders so that the majority of the substance is liquid, with a small proportion in vapour form above the liquid; impurities are present in both phases but in differing proportions. Samples shall be taken from the liquid phase so that they are representative of the majority of the substance in the cylinder, by tilting or inverting the cylinder so that the outlet is at the lowest point.

17. Sampling line and connection

It is preferable for samples to be passed directly from the supply cylinder to the analysis equipment; a sampling line is required for this purpose. The sampling line serves primarily to vapourise the liquid SF_6 coming from the cylinder, and may also conveniently incorporate a fine-control needle

valve since it is often difficult to control flow by means of the cylinder valve alone. The sample line assembly would thus consist of the following components.

17.1 *Fine-control valve (optional)*

An all-metal fine-control needle valve or all-metal pressure reducing valve, screwed directly into the outlet of the cylinder valve or connected to it by a short length of stainless steel tubing similar to the sample line itself (see Sub-clause 17.2).

Note. — The portion of the system connected to the cylinder must be suitable for working at pressures that may occur in the cylinder, that is, up to about 40 bars.

Warning. — The cylinder valve and needle valve must never be left closed at the same time with liquid SF₆ trapped between them, as a rise of temperature is likely to lead to an explosion.

17.2 *Sample line*

The sample line may conveniently consist of approximately 2 m of stainless steel tubing, approximately 3 mm internal diameter. One end is fitted by means of a metal connector to the fine-control valve (or directly to the outlet of the cylinder valve, if no fine-control valve is used); the other end is connected without valves or other restriction to the analysis apparatus or to the intermediate sampling cylinder.

17.3 *Joints*

To make gas-tight threaded joints, polytetrafluoroethylene (PTFE) tape is permissible but a grade containing no plasticiser should be used, otherwise there is a risk of introducing impurities into the sample. Permanent metal-to-metal joints in the sample line assembly are to be brazed or soldered (afterwards being cleaned of flux). Other types of all-metal (e.g., compression) joints may be used.

At the analysis apparatus, the butt-joint technique described in Sub-clause 24.3 is suitable.

17.4 *Use of sample line*

To vapourise the liquid from the cylinder, it may be necessary to warm the sample line slightly. This warming may be by a heating tape or by a small hot-air blower. The line may be coiled for ease of handling.

Purge the sample line by a gentle flow of sulphur hexafluoride for several minutes immediately before beginning a test.

18. **Intermediate sampling cylinder**

If it is impossible to take gas from the supply cylinder and pass it directly into the analysis equipment, it may be necessary to use an intermediate sampling cylinder.

18.1 *Filling technique for sampling cylinders*

Heat the cylinder to 100 °C with its valve open and evacuate it with an oilsealed rotary pump. Connect the evacuated cylinder to the SF₆ container, and, after cooling the cylinder, introduce a small amount of liquid SF₆. Shake the cylinder and vent off the gas. Re-evacuate the cylinder and repeat the washing procedure. Introduce into the cylinder the required sample quantity.

Note. — It must be realized that impurities present in an intermediate cylinder will generally be added to those already in the consignment; thus if the sample is to pass the required purity standards, every effort must be made to ensure that the sampling cylinder is chemically clean, otherwise a higher standard of purity in the original cylinder may well be necessary. (For example, sampling cylinders must not be used for materials other than new SF₆. When not used they should be stored filled with SF₆ under a slight pressure.)

Warning. — Intermediate sampling cylinders must withstand a test pressure of 70 bars and must not be overfilled. In temperate countries a maximum filling ratio of 1.04 kg SF₆ per litre cylinder volume may be allowed. It is recommended that the sampling cylinder be weighed just before and after filling.

SECTION SEVEN — IDENTIFICATION OF GAS

19. General

If there is doubt whether the material supplied is sulphur hexafluoride, identification may be confirmed by the following methods.

20. Infra-red absorption spectrum

Where the required apparatus is available, it may be verified whether the gas is SF₆ by examining its infra-red absorption spectrum and comparing this with that of gas from another source known to be SF₆.

21. Density determination

21.1 Principle of method

A known volume of gas is weighed.

21.2 Apparatus

A glass bulb or cylinder fitted with vacuum-tight stopcocks at opposite ends. A capacity of about 100 ml is suitable but a larger volume may be used provided it can be accommodated on the balance pan and provided its mass is appropriate to the range of the analytical balance.

21.3 Calibration of the volume of the glass bulb

Obtain the volume of the bulb by weighing it to ± 0.1 g empty and then full of distilled water at approximately ambient temperature; the difference gives the mass of water and hence the volume of the bulb at that temperature.

Note. — For borosilicate glasses no further bulb volume correction is required.

21.4 Operating procedure

Carefully dry the bulb, then evacuate it and weigh it to ± 0.20 mg.

Cool the bulb to just below ambient temperature and then fill it with sulphur hexafluoride. Allow the filled bulb to attain ambient temperature then momentarily open a stopcock to bring the gas in the bulb to atmospheric pressure. Note the ambient temperature and barometric pressure and then reweigh the bulb.

Note. — Care should be taken to ensure that the bulb is properly filled. It will be so if the bulb is purged for about 1 min at 5 l/min.

Warning. — In both evacuation and filling with SF₆, proper safety precautions must be taken.

21.5 Calculation of result

$$\text{Density (kg/m}^3\text{)} = 1\,000 \frac{m_2 - m_1}{V}$$

where:

m_2 = mass of bulb filled with SF₆, in grams

m_1 = mass of evacuated bulb, in grams

V = volume of gas, corrected to reference temperature and pressure (e.g. 20 °C and 760 torr) from the temperature and pressure existing at the time the pressure in the bulb was adjusted, in millilitres

SECTION EIGHT — TOXICITY

22. Toxicity tests

22.1 Experimental details

Five female albino mice, previously maintained under barriered conditions so that they are as free as possible from disease, are to be used for each test. They are to be kept under observation for at least 5 days prior to exposure to ensure that they are in good health. They are then to be introduced into the exposure chamber containing an atmosphere of 79 % SF₆ and 21 % O₂ by volume and kept there for a period of 24 h. During this time water and laboratory mouse diet are to be supplied *ad libitum*.

22.2 Exposure conditions

The SF₆ is taken from a cylinder maintained in an inverted position so that the gases generated are truly representative of the liquid phase. The oxygen is supplied from a cylinder and the required ratio of 79 % SF₆: 21 % O₂ by volume is to be obtained by balancing the rates of flow of the two gases as measured by calibrated flow-meters. The two gases are to be mixed in a mixing vessel prior to being led into the exposure chamber.

The rate of gas flow must never be less than $\frac{1}{8}$ th of the total volume of the exposure chamber per minute.

After 2 h (when stable conditions have been obtained) and 22 h (to ensure that no significant deviation from the required values has occurred), the atmosphere is to be analysed for oxygen content by means of Dräger oxygen analysis tubes or other suitable means.

22.3 Observations

During exposure the mice are to be observed at frequent intervals for evidence of abnormal behaviour. On completion of the exposure period, they are to be removed and returned to their original cages for subsequent observation during a period of 72 h.

22.4 Interpretation of results

Any deviation from normal behaviour on the part of the mice must be viewed with suspicion and may or may not indicate the presence of toxic impurities. If frank symptoms of toxicity are observed then the response must be considered positive.

It is conceivable that one or more animals may occasionally exhibit signs of unusual reaction, or indeed may die either during exposure or during the observation period, perhaps from causes unrelated to the experimental treatment. In such cases the experiment should be repeated with a total of ten mice to confirm or deny the real significance of the previous observation.

Any mouse that dies during the investigation and/or any mouse exhibiting any deviation from normal behaviour should be subjected to detailed autopsy in an attempt to establish the cause.

SECTION NINE — WATER CONTENT AND CONDENSATION TEMPERATURE

23. General

In general the major item of condensable material in new SF_6 will be water and several methods may be used to verify that the water content does not exceed the permitted amount. However, the gravimetric method is the only absolute method and therefore must be available to check other methods. The gravimetric method shall be used as the reference method in cases of doubt or dispute for the determination of the water content of the gas. A suitable technique is described in Clause 24.

For the routine assessment of water content it may be convenient to use either an electrolytic or a condensation temperature technique and suitable procedures are specified in Clauses 25 and 26. These tests are such that they can be performed quite readily by a purchaser of new gas.

The condensation temperature technique, however, is not specific to water and will reveal, within the limits of detection, any condensable material that is present in the new gas.

In certain items of equipment, it may be necessary to avoid the condensation of any materials and in these circumstances a maximum condensation temperature may be specified by agreement between the supplier and purchaser, either as an additional test requirement or as an alternative to the water content requirement of Table I.

If it is established that the condensation temperature of the gas under test is below -42°C then the gas may be assumed to comply with the requirements for water content given in Table I (water content not more than 15 ppm by mass).

If, however, the condensation temperature is above -42°C , it cannot be assumed that the condensing material is solely water and if the actual water content is to be determined either the gravimetric technique described in Clause 24 or the electrolytic method of Clause 25 must be used.

Note. — The relationship between condensation temperature and water content for new gas assuming no other condensable material is present can be calculated from standard tables.

24. Gravimetric method of water determination (for reference purposes)

24.1 Principle of method

A measured volume of sulphur hexafluoride is passed through tared tubes containing magnesium perchlorate (anhydron) as desiccant. The increase in mass of the tubes gives the water content of that volume of gas.

Due to the inherent difficulties of the method, the test should be carried out only by skilled personnel. Changes of ambient conditions during the test, such as temperature and humidity, may influence the results. It is advisable therefore to operate in an air-conditioned room.

24.2 Apparatus

Desiccant tubes: three glass or stainless steel U-tubes A_1 , A_2 and A_3 , approximately 100 mm high and 13 mm bore containing an intimate mixture of two volumes of magnesium perchlorate and one volume of glass powder, $355\mu\text{m}$ - $420\mu\text{m}$. Immediately after filling the tubes, insert glass wool plugs to keep the desiccant in place and then seal off the ends of the tubes with a blow-pipe flame. Alternatively polychloroprene caps can be used. (See Figure 3.)

Note. — While the tubes contain desiccant but are not connected to the absorption train, the side-arms should be closed by means of polychloroprene caps, etc. When not in use the caps must be kept in dry conditions.

Alternatively the inlet and outlet arms of the tubes may be fitted with stopcocks which will help in isolating the desiccant from the atmosphere when the caps must be removed for weighing or installation in the absorption train.

24.2.1 *Guard tube*

A guard tube containing the same desiccant as in tubes A₁, A₂ and A₃.

Note. — This guard tube protects A₃ from water contamination coming from the gas meter.

24.2.2 *Gas meter*

A gas meter to measure a quantity of 50 l or more, to an accuracy of $\pm 2\%$.

24.2.3 *Filter*

A fritted glass filter, porosity 2 (40 μm to 50 μm).

24.2.4 *Supply of dry oil-free air*

This may be prepared by passing air through a vessel packed with layers of soda-asbestos and magnesium perchlorate.

Note. — Soda-asbestos is a mixture of caustic soda (NaOH) and asbestos, often used as an absorbent for CO₂.

25.2.5 *Stopcocks (optional)*

Isolating stopcocks as shown in Figure 3, are useful to maintain dry conditions in the absorption train when the desiccant tubes are removed for weighing.

24.3 *Operating procedure*

Dry the inlet end of the absorption train, including the filter and any isolating stopcocks, with hot air and then connect tubes A₁, A₂ and A₃. Polychloroprene tubing shall be used for the connections. It is essential that the components of the absorption train are butt-jointed; the flexible tubing shall be reasonably tightly fitting and act as sleeving.

Open the stopcocks and pass dry air through the absorption train at the rate of about 250 ml/min for 15 min to condition the tubes. Close the stopcocks, disconnect tubes A₁, A₂ and A₃, and close the side arms of all tubes with polychloroprene caps.

Carefully wipe the tubes with a soft dry cloth or chamois leather and place them in the balance case.

Note. — Once the tubes have been wiped, subsequent handling must be with dry tongs or with chamois leather finger stalls, etc. The glassware should also be protected from dust and other contamination.

Allow the tubes to stand in the balance case for 20 min, then weigh them separately to the nearest 0.1 mg. The caps should be removed temporarily from the side-arms of the tubes.

The conditioning procedure shall be repeated until each single tube shows a difference in mass less than 0.2 mg between two consecutive weighings, in order to ensure complete conditioning.

Dry the sampling line with hot air and then purge the sampling line with sulphur hexafluoride. Reconnect tubes A_1 , A_2 and A_3 to the absorption train. Note the gas-meter reading, the temperature at the gas meter and the atmospheric pressure. Open the stopcocks in the absorption train and, with a flow-rate of about 250 ml/min, pass about 50 l to 100 l of sulphur hexafluoride gas through the train.

Note. — If a wet gas meter is used, the indicated volume of gas will be the volume of dry gas plus the volume of water vapour needed to saturate it at the temperature and pressure at the meter.

Close the cylinder valve and needle valve, close the stopcocks in the absorption train and note the new reading on the gas meter, the temperature at the meter and the atmospheric pressure. Now pass dry air through the absorption train to displace the sulphur hexafluoride gas in the desiccant tubes. Disconnect tubes A_1 , A_2 and A_3 from the absorption train and refit the caps to the side-arms. Wipe and reweigh the tubes as before.

If, in any single determination, tube A_2 shows an increase in mass greater than 1 mg or 10% of the increase in mass of A_1 , both these tubes should be refilled with fresh desiccant and the test repeated. No water should pass through the train as far as tube A_3 so that if there is any measurable increase in the mass of this tube, tubes A_1 and A_2 should be refilled with fresh desiccant and the test repeated.

24.4 Calculation of result

Calculate the corrected volume V_c of SF_6 at 20 °C and 760 torr as follows:

$$V_c = \frac{\frac{1}{2} (p_1 + p_2) 293}{760 (273 + \frac{1}{2} (t_1 + t_2))} (V_2 - V_1)$$

where:

p_1 and p_2 = initial and final atmospheric pressures at the gas meter, torr
 t_1 and t_2 = initial and final temperatures at the gas meter, Celsius degrees
 V_1 and V_2 = initial and final gas meter readings, litres

Then the water content as ppm (mass by mass) is:

$$\frac{(m_x - m_a) + (m_y - m_b)}{6.16 V_c} 1\,000$$

where:

m_a = mass of tube A_1 after conditioning, in milligrams
 m_b = mass of tube A_2 after conditioning, in milligrams
 m_x = mass of tube A_1 at the end of the determination, in milligrams
 m_y = mass of tube A_2 at the end of the determination, in milligrams
 V_c = volume of SF_6 used at 20 °C and 760 torr, in litres

25. Electrolytic method (for routine purposes)

25.1 Principle of method

The essential basis of the electrolytic hygrometer is a cell through which a water-containing gas is passed.

This cell contains two rhodium-wire electrodes coiled inside or on the outside of an insulating tube or former. The small space between these electrodes is bridged by a thin film of phosphorous pentoxide (P_2O_5). A direct voltage is applied between these electrodes, but so long as the P_2O_5 is completely dry, no current flows.

Gas to be analysed is passed through the cell close to the P_2O_5 , which absorbs any water present in it. Simultaneously, current passes through the film, electrolyzing the absorbed water into hydrogen and oxygen; the amount of electricity needed is a measure of the amount of water absorbed, from Faraday's law. If the rate of gas flow is steady and accurately known, the electrical current flowing when steady-state conditions have been reached is a measure of the water concentration in the gas; this current may be measured by a sensitive ammeter. This is the normal method of use but in circumstances in which steady-state conditions are not achieved, the current may be recorded using a potentiometer recorder so that the energy may be calculated by integration.

25.2 Apparatus

Several manufacturers offer apparatus of this type and any one may be used for the present purposes. Preference may be given to a type in which the wire electrodes are accessible so that recoating with phosphoric acid is practicable.

All-metal joints shall be used to connect the apparatus to the sampling line.

Overall accuracy of the equipment involves absorption efficiency, flow meter accuracy and ammeter accuracy. The absorption efficiency especially may lead to a large error without any obvious indication that this has occurred.

This is most likely if the instrument has been used for purposes other than the examination of new SF_6 , when it may have become saturated with water, contaminated with oil vapour, etc. Where an electrolytic instrument is used for many purposes, only by very frequent recalibration can the accuracy of observation be assured.

Where an instrument is used only for examination of new SF_6 , overall accuracy is to be assessed for each instrument at least every 12 months by comparison with results from gravimetric tests using SF_6 containing not more than 15 ppm by mass of water; an accuracy of ± 2 ppm at about this water content shall be obtained. In addition to the above periodical recalibration each instrument may with advantage be checked more frequently by reference to a cylinder of SF_6 kept for that purpose. It is acceptable to use nitrogen having the same concentration of water per unit volume (i.e., not more than 80 ppm of water by mass).

26. Dew point method

Under consideration.

SECTION TEN — CARBON TETRAFLUORIDE, OXYGEN AND NITROGEN

27. Principle of method

Samples of SF_6 gas are analysed by gas-solid chromatography using a katharometer detector and an adequate separating column. The concentration of air (or its constituents: oxygen and nitrogen) as well as carbon tetrafluoride are determined from their peak areas and correction factors which take into account the differences of detector response to the compounds under investigation.

28. Apparatus

28.1 Gas chromatograph with thermal-conductivity detector

Any instrument with thermal-conductivity detector suitable for use with columns about 2 m long and about 5 mm or 6 mm outside diameter can be used. The oven shall be capable of being maintained

at 40 °C. The gas samples shall be injected by means of a properly designed injection valve capable of injecting 1 ml samples. Injection of samples by hypodermic syringe shall not be permitted.

28.2 Recorder

This must suit the actual gas chromatograph used but should preferably be with a 0 mV to 1 mV range, 1 s response time and a 250 mm wide chart.

28.3 Carrier gas

Helium is preferred as carrier gas. Hydrogen may be used but the necessary safety precautions must be taken in that case.

28.4 Separating column

The column recommended for the determination of ($O_2 + N_2$) and CF_4 is a 2 m long stainless steel tubing packed with 30-50 mesh silicagel coated with 3% of 2 ethyl-hexyl sebacate. Packed columns are supplied by several manufacturers or may be prepared according to manufacturer's instructions.

New columns should be conditioned before use at 120 °C for at least 4 h while passing carrier gas through them.

Other column packing may be used for special purposes. Molecular sieves allow separate determinations of oxygen and nitrogen (see figure 4, page 42). Crosslinked polystyrene, 80-100 mesh will enable besides air and CF_4 also the determination of CO_2 as well as lower sulphur fluorides (such as SOF_4 , SO_2F_2 , $SOF_2 + SF_4$, C_2F_6) if a sufficient concentration (at least 1%) is present.

Note. — For information, 13X molecular sieve and cross-linked polystyrene such as Porapak Q have been found suitable.

29. Procedure

Switch on the gas chromatograph and, operating according to the manufacturer's instructions, allow it to stabilize using an oven temperature of 40 °C and a carrier gas flow-rate set within the range 50 ml/min to 80 ml/min.

Attach a piece of 3 mm bore tubing, about 50 cm long, to the outlet of the sampling loop of the injection valve. Purge the sampling line from the cylinder with SF_6 gas and then connect the sampling line to the inlet of the sampling loop. Flush all air or carrier gas out of the loop with SF_6 gas from the cylinder to be analysed, then close the needle valve on the sampling line and operate the injection valve on the gas chromatograph in that order with the aim of bringing the gas in the sampling loop to atmospheric pressure.

Maintain steady conditions to obtain the chromatogram. Components are eluted in the following order: air, CF_4 , SF_6 .

30. Calibration

The recorded peak areas of the different components are not directly proportional to the corresponding concentrations by mass in the mixtures, owing to the differences in detector response to individual components.

Empirical correction factors must be determined and the recorded peak areas multiplied by them. The most accurate determination of the correction factors is obtained by analysing standard mixtures of air and carbon tetrafluoride with sulphur hexafluoride.

Calibration mixtures may be prepared by dynamic mixing of main and impurity components *

From the chromatograms of the standard mixtures the correction factors, f_{air} and f_{CF_4} , are calculated for air and CF_4 relative to SF_6 , for which $f_{\text{SF}_6} = 1$ can be set.

A very much simpler, although slightly less accurate method for determining the correction factors involves injections of 1 ml samples of pure constituents into the chromatographic column under the same analytical conditions.

The correction factor f_x of the component x with respect to SF_6 is given by the relation:

$$\text{SF}_6 \text{ peak area} = f_x \frac{M_{\text{SF}_6}}{M_x} \cdot x \text{ component peak area}$$

where:

M_{SF_6} = molecular mass of $\text{SF}_6 = 146$

M_x = molecular mass of component x
(28.8 for air, 88 for CF_4)

The following correction factors may be used for routine analysis:

$$\begin{aligned} f_{\text{SF}_6} &= 1.0 \\ f_{(\text{O}_2 + \text{N}_2)} &= 0.4 \\ f_{\text{CF}_4} &= 0.7 \end{aligned}$$

31. Results

Obtain the area of each peak by multiplying the peak height by the peak width at half height.

Correct the measured area for differences in detector response by multiplying it by the relevant correction factor.

The percentage by mass of any component is then given by:

$$x = \frac{A_x}{A_t} 100$$

where:

x = mass percent of component x

A_x = corrected area of the peak for component x (air or CF_4)

A_t = sum of the corrected areas of peaks (air, CF_4 and CF_6)

SECTION ELEVEN — HYDROLYSABLE FLUORIDES

32. Principle

The gas is shaken in a large gas pipette with dilute sodium hydroxide solution. The fluoride ion produced is determined colorimetrically with lanthanum alizarin complexone.

* The method described by J. C. Cage (Journal of Scientific Instruments 30 (1953) 25) is recommended. In this method a small flow of the impurity is delivered at a known rate from a syringe, whose piston is driven by a synchronous motor and suitable gear train, into a stream of SF_6 flowing at a known steady rate. The flow rates of the two streams are adjusted to give the impurity concentration required.

33. Apparatus

Gas pipette 1 l capacity with tubulure fitted with a serum cap.

Hypodermic syringe 10 ml capacity.

Mercury seal.

Spectrophotometer with 2 cm or 4 cm cells.

34. Reagents

- a) *alizarin fluorine blue* (3-aminomethylalizarin-NN-diacetic acid);
- b) *ammonium hydroxide solution* density 0.880 g/ml;
- c) *ammonium acetate solution*, 20 % mass/volume;
- d) *anhydrous sodium acetate*;
- e) *glacial acetic acid*;
- f) *acetone*;
- g) *lanthanum oxide*;
- h) *hydrochloric acid*, 0.1 N;
- i) *hydrochloric acid*, 2 M dilute 17.2 ml of hydrochloric acid (density 1.18 g/ml) to 100 ml with water
- j) *sodium fluoride*;
- k) *sodium hydroxide solution*, 0.1 N;
- l) *lanthanum-alizarin complexone reagent*;

Dissolve 0.048 g of alizarin fluorine blue a) in 0.1 ml of ammonium hydroxide solution b) and 1 ml of ammonium acetate solution c) together with a few millilitres of water. Filter the solution through a small Whatman No. 1 paper into a 250 ml graduated flask containing 8.2 g of anhydrous sodium acetate d) and 6.0 ml of glacial acetic acid e) dissolved in just sufficient water. Wash the filter with a small quantity of water and then add to the flask 100 ml of acetone, slowly with swirling.

Dissolve 0.041 g of lanthanum oxide g) in 2.5 ml of 2 M hydrochloric acid i) warming gently to aid dissolution. Mix the warm solution with the aqueous acetone solution in the flask and dilute the solution to 250 ml with water. Mix the solution thoroughly and when it is cool, adjust the volume to 250 ml.

This reagent is stable for one week.

- m) *stock sodium fluoride solution*, 1 mg F/ml;

Dissolve 2.210 g of dried sodium fluoride j) in 50 ml of water containing 1 ml of 0.1 N sodium hydroxide solution and dilute to 1 l with water.

Store in a polyethylene bottle.

- n) *standard sodium fluoride solution* 10 µg F/ml;

Dilute the stock sodium fluoride solution j) 100 — fold daily as required.

35. Procedure

Evacuate the gas pipette. Connect the gas sample point with a T-piece one arm of which is connected to a mercury seal. Open the sample valve and allow a steady flow of gas to bubble through the seal. Fill the sample bulb by cautiously opening the tap so that gas flows into the bulb, observing the seal to ensure that no mercury is sucked into the pipette.

The pipette is full when the gas again bubbles freely into the seal. Disconnect the pipette and rapidly open and close the tap to equalize pressure to the atmosphere. Using the hypodermic, inject 10 ml 0.1 N sodium hydroxide solution into the bulb. Shake for 1 min in every 5 min for a period of 1 h.

Transfer the solution from the bulb to a small beaker, adjust to pH 5 with 0.1 N hydrochloric acid solution *h*) and transfer to a 100 ml graduated flask. Add 10 ml lanthanum-alizarin complexone reagent *l*). Dilute to the mark with water, mix and allow to stand for a 30 min away from direct light. Measure the optical density in a 2 cm or 4 cm cell at 600 nm against a " blank " carried out upon all the reagents including the 10 ml 0.1 N sodium hydroxide solution *k*).

Read the amount of fluorine from a calibration curve. It is advisable to prepare a fresh calibration curve daily.

36. Preparation of calibration curve

To 10 ml portions of 0.1 N sodium hydroxide solution *k*) add respectively 0 ml, 0.5 ml, 1 ml, 1.5 ml and 2.0 ml portions of the 10 µg/ml standard fluoride solution *n*). Adjust to pH 5 with 0.1 N hydrochloric acid solution *h*), transfer to 100 ml volumetric flasks and continue as described above.

37. Calculation of result

Calculate the amount of hydrolysable fluorides as ppm (mass by mass) of hydrofluoric acid (HF) as follows:

$$\text{HF (ppm)} = \frac{20\,000\,n}{19 \times 6.16\,V \frac{p}{760} \frac{293}{273 + t}}$$

where:

n = amount of fluorine found in the form of fluorides, in micrograms

V = volume of the gas pipette, in millilitres

p = atmospheric pressure, in torr

t = ambient temperature, in Celsius degrees.

SECTION TWELVE — ACIDITY

38. Principle of method

Acid and acid producing constituents in the sample are absorbed in standard alkali solution and the excess alkali back-titrated with standard acid.

The resultant acidity is expressed as HF.

39. Apparatus

39.1 Gas wash bottles

Drechsel type, 250 ml capacity. Three of these are required, one fitted with a fritted glass gas distributor and two with an open tube distributor.

39.2 Microburette

Graduated to 0.01 ml.

39.3 Gas meter

To measure about 10 l or more of sulphur hexafluoride gas to an accuracy of $\pm 2\%$.

40. Reagents

40.1 Bromocresol purple indicator solution

Dissolve 1 g of bromocresol purple in 100 ml of ethanol (95%).

Note. — Strictly speaking this indicator is not particularly suitable for weak acid titrations. However bromocresol purple is preferred in practice because:

- a) the colour change is very distinct;
- b) in general it is not necessary to prevent contact with atmosphere during titration;
- c) the present test is solely for go/no go purposes and extreme accuracy is therefore not required.

40.2 Standard sodium hydroxide solution 0.01 N

This reagent should be carbonate-free.

40.3 Standard sulphuric acid solution 0.01 N

41. Procedure

Boil about 600 ml water for 5 min in a 1 l flask, then cool it quickly to room temperature. Add about 10 drops of bromocresol purple indicator solution, and then just neutralize the water by adding standard sodium hydroxide solution (0.01 N) from a microburette.

Pour about 150 ml of the neutralized solution into each of the three Drechsel bottles and then add to each from a microburette, 2.00 ml of sodium hydroxide solution (0.01 N). Replace the heads on the bottles and keep one of the open-tubed bottles as a blank.

Assemble the gas washing train as shown in Figure 5, keeping the fritted glass washing bottle first of the two in the train.

Note. — The object of the plain-tubed gas wash bottle is to trap any spray from the fritted glass bottle.

Purge the sampling line with SF_6 , then close the needle valve. Note the gas-meter reading. Connect the sampling line to the train and, by adjusting the needle valve, pass gas through the train at the rate of about 0.5 l/min for about 20 min.

Notes 1. — Too fast a flow rate can cause foaming. The fastest flow rate that can be used must be found by trial; the suggested 0.5 l/min is given as a guide.

2. — A bigger sample—for instance 50 l/min—should be used if a better accuracy is needed.

Stop the flow of gas and note the new reading on the gasmeter.

Disconnect the gas wash bottles and titrate the solutions in each of the three bottles with standard sulphuric acid solution (0.01 N).

42. Calculation of result

Calculate the corrected volume V_c of SF_6 used at 20 °C and 760 torr as follows:

$$V_c = (V_2 - V_1) \frac{\frac{1}{2} (p_1 + p_2)}{760} \frac{293}{273 + \frac{1}{2} (t_1 + t_2)}$$

where:

V_c = corrected volume, in litres

p_1 and p_2 = initial and final atmospheric pressures at the gas meter, in torr

t_1 and t_2 = initial and final temperatures at the gas meter, in Celsius degrees

V_1 and V_2 = initial and final readings of the gas meter, in litres

Calculate the acidity as ppm (mass by mass) of hydrofluoric acid (HF) by means of:

$$\text{HF} = \frac{200 (B - X) + (B - Y)}{6.16 V_c}$$

where:

B = the volume of sulphuric acid solution used for the titration of the blank solution, in millilitres

X = the volume of sulphuric acid solution used for the titration of the solution in wash bottle 1, in millilitres

Y = the volume of sulphuric acid solution used for the titration of the solution in wash bottle 2, in millilitres

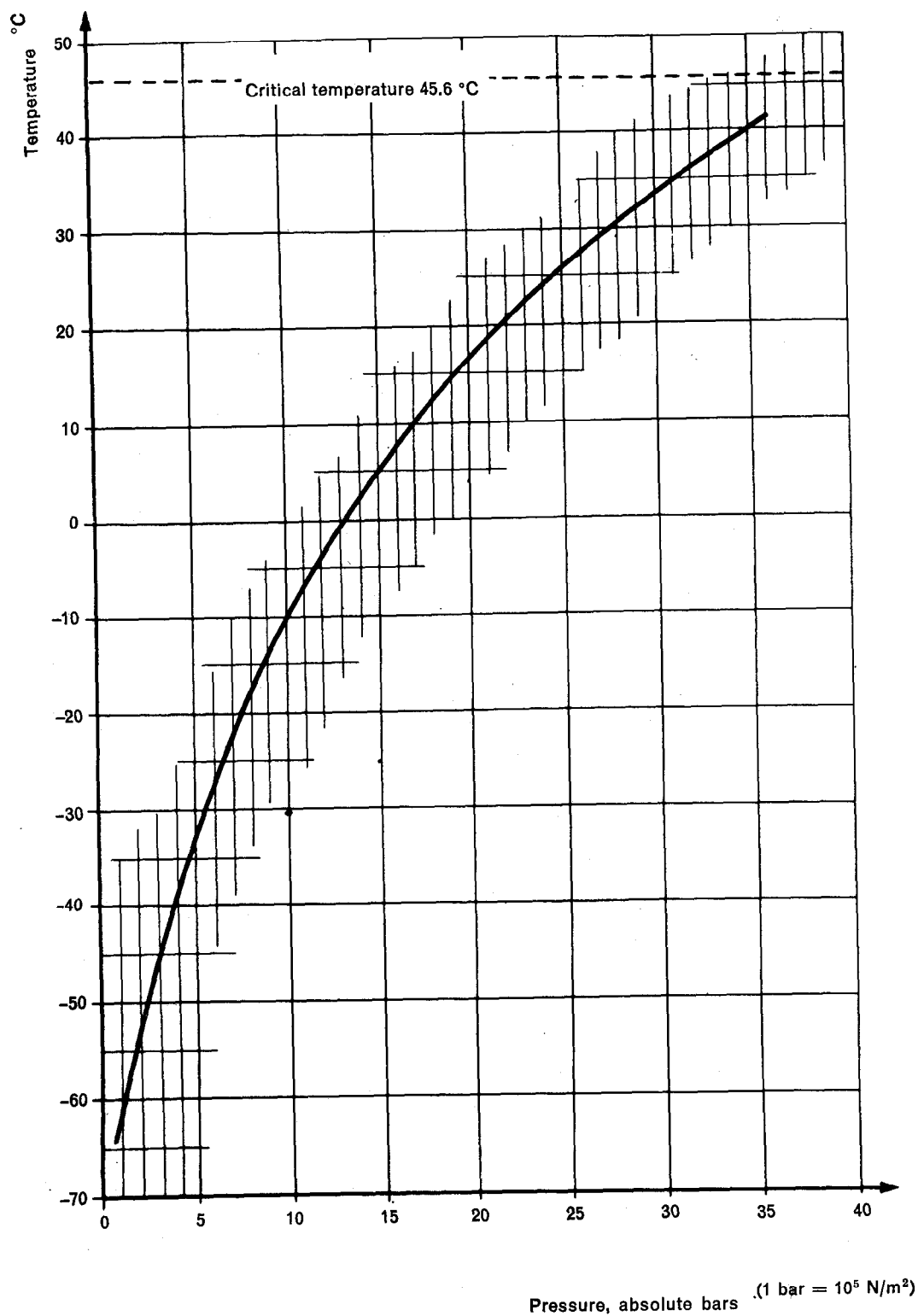
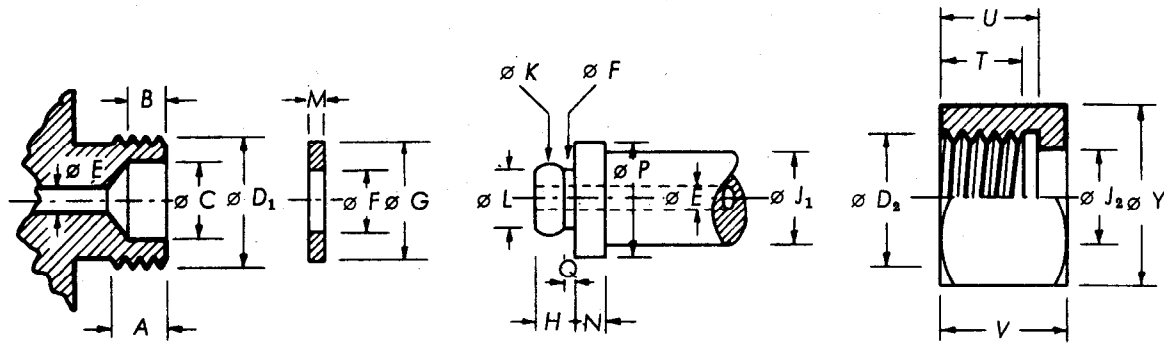


FIG. 1. — Saturated vapour pressure curve for SF_6 .



Dimension	mm	Dimension	mm	Dimension	mm
<i>A</i>	9 min	$\varnothing L$	1	$\varnothing D_2$	21.8
<i>B</i>	7	$\varnothing K$	12	$\varnothing J_2$	16.3 - 16.1
$\varnothing C$	13.3 - 12.7	$\varnothing P$	19	<i>T</i>	12 min
$\varnothing D_1$	21.780 - 21.387	$\varnothing J_1$	16.0 - 15.8	<i>U</i>	16.3 - 16.0
$\varnothing E$	6	<i>H</i>	6	<i>V</i>	21
$\varnothing F$	11.5	<i>N</i>	5	$\varnothing Y$	30
$\varnothing G$	18.0 - 18.5	<i>Q</i>	1.8		
<i>M</i>	2	Threads $\varnothing D_1$ and $\varnothing D_2$ = 14 threads per inch right-hand whitworth form			

FIG. 2. — Valve outlet.

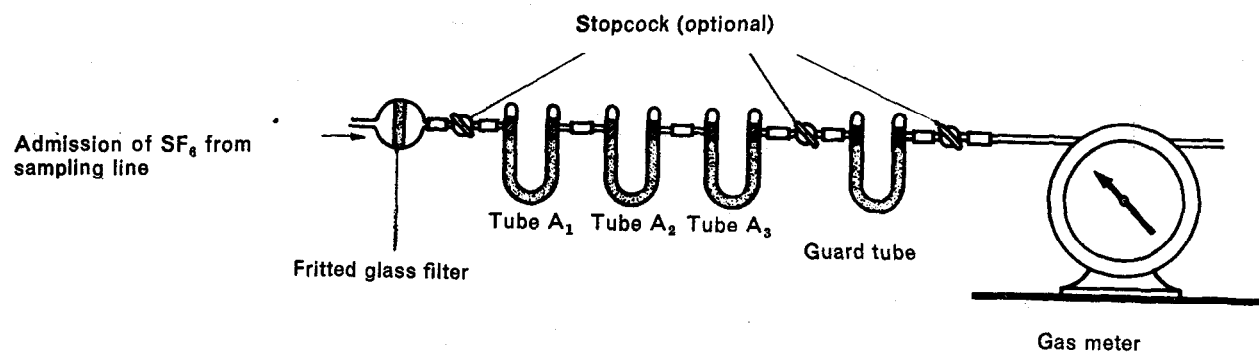
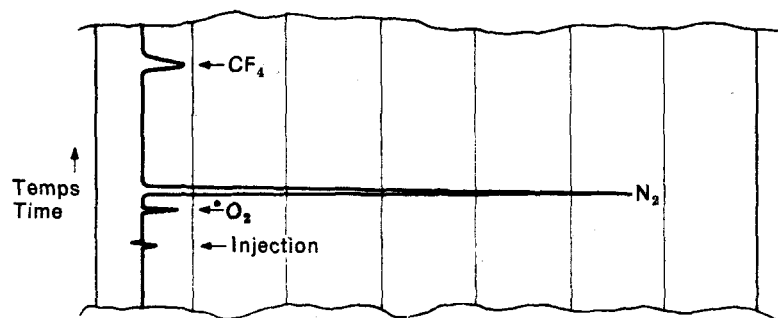


FIG. 3. — Gravimetric determination of water in SF_6 gas.



Chromatogram obtained using a molecular sieve column and helium carrier gas; showing order of elution of O_2 , N_2 and CF_4

Chart speed approximately $\frac{1}{2}$ cm/min ($\frac{1}{4}$ in/min)

FIG. 4. — Example of chromatogram obtained when determining oxygen, nitrogen and carbon tetrafluoride.

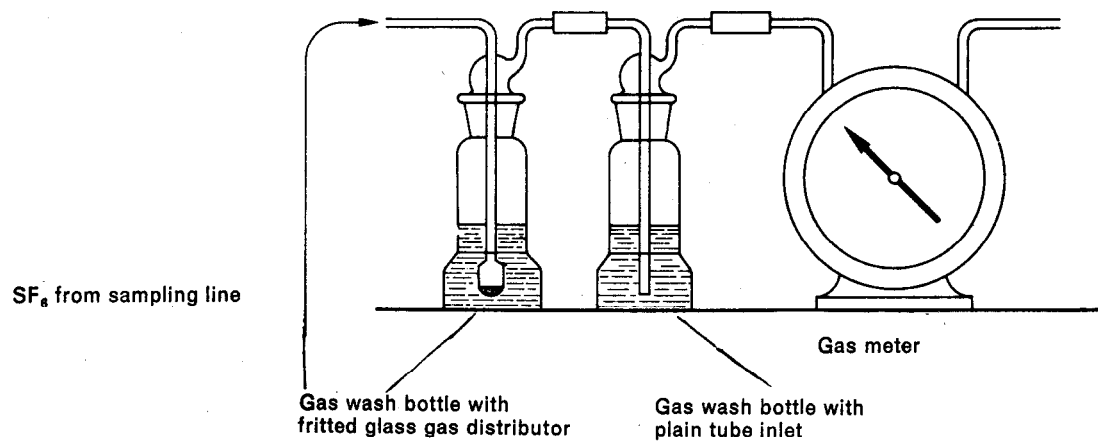


FIG. 5. — Apparatus for the determination of free acidity.

SECTION THIRTEEN — MINERAL OIL CONTENT

43. Scope

This method covers the determination of the amount of mineral oil in new sulphur hexafluoride. It does not apply, as written, to the determination of synthetic lubricants.

44. Principle of the method

Mineral oil dispersed in sulphur hexafluoride is absorbed in carbon tetrachloride by bubbling a known volume of gas through the solvent contained in two absorption tubes. The concentration of mineral oil in carbon tetrachloride is then determined by the infrared absorbance of the solution at 2930 cm^{-1} corresponding to the absorption band of paraffinic methyl and methylene groups.

Note. — Carbon tetrachloride is highly toxic and health regulations of many countries prohibit its industrial use. However, carbon tetrachloride is widely used in analytical laboratories, especially in connection with infrared spectroscopy. Even where local legislation prohibits the laboratory use of carbon tetrachloride the method described below can be followed by replacing carbon tetrachloride with less toxic tetrachloroethylene. In this case, on account of the absorption of tetrachloroethylene itself in the region of 2930 cm^{-1} , the accuracy of the method is decreased, especially for lower oil contents. The estimate of precision given in Clause 49 only applies to tests performed with carbon tetrachloride.

45. Apparatus

45.1 Infrared spectrophotometer

Any suitable infrared spectrophotometer.

45.2 Absorption cells

Two types of fixed cells for liquids provided with sodium chloride windows may be used:

Type 1: with a sample path length of at least 3 mm, is suitable for use with Procedure A;

Type 2: with a sample path length of at least 20 mm, is suitable for use with Procedure B.

45.3 Sampling line

The sampling line, which connects the absorption train to the gas cylinder, shall be in accordance with Clause 17.

All parts of the sample line must be suitable for rinsing with carbon tetrachloride (or tetrachloroethylene) as described in Sub-clause 47.2.1. For this reason, to provide fine control of gas flow, an all-metal needle valve is preferred to a pressure reducing valve (see also the note under Sub-clause 45.4).

45.4 *Absorption train*

The absorption train consists of two gas wash bottles, 100 ml capacity, made of borosilicate glass and dimensioned as in Figure 6, page 14. Ground joint Drechsel-type absorption tubes are also allowed provided that they can be used without any sealing grease. The inlet tubing is fitted at its end with a fritted glass disk of porosity Grade 1 (average pore diameter 90 μm to 150 μm). Connections to the sampling line and between the components of the absorption train shall be butt-jointed, the flexible tubing used as sleeving being made of silicone rubber or fluorinated elastomers.

Note. --- To check the suitability of the tubing used as sleeving, immerse a 3 cm long specimen in 50 ml of carbon tetrachloride for 30 min. Obtain the absorbance of the washing liquid at 2930 cm^{-1} against unused solvent. No absorption should be detected.

45.5 *Gas-meter*

A gas-meter to measure 30 l of SF_6 gas to an accuracy of $\pm 2\%$.

45.6 *Evaporating crucible*

A borosilicate glass or porcelain crucible, capacity 50 ml to 250 ml.

45.7 *Graduated flasks*

One 10 ml and one 100 ml borosilicate glass graduated flask.

46. **Reagents**

46.1 *Carbon tetrachloride*

Carbon tetrachloride, analytical grade, freshly distilled (boiling point 76-77 °C).

46.2 *Sodium sulphate*

Sodium sulphate, anhydrous.

46.3 *Tetrachloroethylene (optional)*

Tetrachloroethylene, analytical grade (boiling point 120-121 °C).

47. **Procedure**

47.1 *Preparation of the calibration curve*

Prepare a number of standard solutions of paraffinic mineral oil per litre of carbon tetrachloride containing approximately 10 mg, 20 mg, 50 mg, 100 mg and 200 mg. The oil should be weighed to the nearest 0.2 mg.

Transfer the standard solutions to the cell of the desired path length and read the absorbances at 2930 cm^{-1} . Repeat the measure on the carbon tetrachloride. Plot the net absorbance values against the concentrations of oil to obtain the calibration curve.

47.2 *Determination of oil content*

47.2.1 *Removal of oil from SF_6 gas*

Add 35 ml of carbon tetrachloride to each of the two clean and dry wash bottles. Carefully rinse the pipework, the valve and the connections with carbon tetrachloride and assemble the sampling line and the absorption train as indicated in Figure 7.

Place the wash bottles in a cooling bath at 0 °C made up with ice and water. Check that the needle valve V_1 is closed and open the main valve on the cylinder. Note the gas-meter reading, the temperature at the gas-meter and the atmospheric pressure.

Carefully open and adjust valve V_1 to allow the gas to flow through the gas wash bottles at a slow steady rate of 10 l per hour maximum.

Note. — If Drechsel-type wash bottles are used, check the tightness of the ground joint by sinking it into the cooling bath.

After passing about 30 l of SF_6 through, close the main valve and continue bubbling as long as the flow continues by itself.

Close valve V_1 and record the new reading of the gas-meter (to the nearest 0.2 l), the temperature at the gas-meter and the atmospheric pressure.

Remove the cooling bath, disconnect the wash bottles and quantitatively transfer the liquid into a 100 ml graduated flask.

Separately rinse the wash bottles and all pipework and connections with 10 ml portions of carbon tetrachloride. Add the washings to the graduated flask and make up to the 100 ml mark with carbon tetrachloride.

Measure the absorbance of the solution at 2930 cm^{-1} in accordance with either Sub-clause 47.2.2 or 47.2.3.

47.2.2 *Infrared determination. Procedure A (3 mm length cell)*

Transfer the solution from the graduated flask to the crucible and evaporate it to a volume of about 5 ml.

Notes 1. — As an alternative, the solution may be concentrated by distillation. In this case, no grease shall be used on the ground joints of the equipment. PTFE sleeves may be used.

2. — Remove water visible on the surface of the solution after the concentration, if any, by adding 1-2 g of anhydrous sodium sulphate. Filter the solution and wash the residue on the filter with a few ml of distilled carbon tetrachloride to avoid any retention of oil.

Quantitatively transfer the residue to a 10 ml calibrated flask. Rinse the crucible with 3 ml of carbon tetrachloride, add the washings to the residue and make up to the mark with carbon tetrachloride.

Fill the 3 mm cell (Type 1) with the solution by means of a pipette and read the absorbance at 2930 cm^{-1} .

Run a blank by evaporating 100 ml of pure carbon tetrachloride (tetrachloroethylene) to 5 ml and measure the absorbance of the residue.

Calculate the concentration of oil from the calibration curve.

47.2.3 *Infrared determination. Procedure B (20 mm path length cell)*

By means of a pipette transfer the solution contained in the 100 ml graduated flask into the Type 2 test cell having a path length of at least 20 mm. Read the absorbance at 2930 cm^{-1} .

Measure the absorbance of the solvent in the same cell at 2930 cm^{-1} .

Obtain the concentration of mineral oil in the solution from the calibration curve.

48. Calculation of results

Calculate the corrected volume V_c at 20 °C and 760 torr, as follows:

$$V_c = \frac{\frac{1}{2}(p_1 + p_2) 293}{760 \left[273 + \frac{1}{2}(t_1 + t_2) \right]} (V_2 - V_1)$$

where:

p_1 and p_2 = initial and final atmospheric pressures (torr);

t_1 and t_2 = initial and final temperatures at the gas-meter (degrees centigrade);

V_1 and V_2 = initial and final gas-meter readings (litres).

Calculate the amount of mineral oil present in the sample of sulphur hexafluoride in parts per million (ppm), as follows:

Procedure A (Sub-clause 47.2.2)

$$C = \frac{(a - b) 10}{6.16 V_c}$$

Procedure B (Sub-clause 47.2.3)

$$C = \frac{(a - b) 100}{6.16 V_c}$$

where:

C = ppm by mass of mineral oil in SF_6 ;

a = concentration in mg/l of mineral oil in the solution;

b = concentration in mg/l of mineral oil in the solvent (blank determination);

V_c = corrected volume (litres) of SF_6 gas used in the test.

49. Sensitivity and precision

Procedure A allows the determination of 0.1 mg of mineral oil dissolved in 10 ml of carbon tetrachloride with a precision of $\pm 25\%$.

Procedure B allows the determination of 0.1 mg of mineral oil in 100 ml carbon tetrachloride to the same precision.

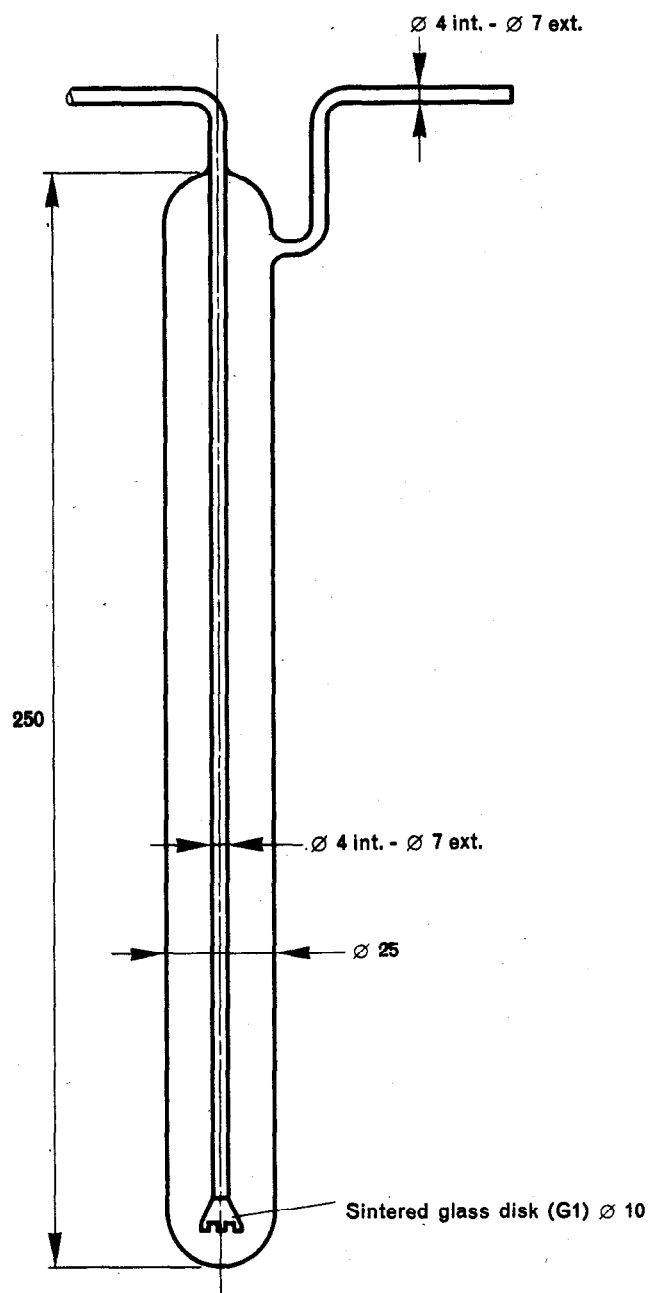
From the above sensitivity and the amount of SF_6 used (30 l), it is possible to calculate that 0.5 ppm by mass of oil in SF_6 may be determined according to either procedure with a precision of $\pm 25\%$.

The precision of the method is better when the amount of oil collected is greater, as follows:

Amount of oil (mg)	Precision
0.1	$\pm 25\%$
0.5	$\pm 15\%$
1.0	$\pm 10\%$

50. Permissible concentration

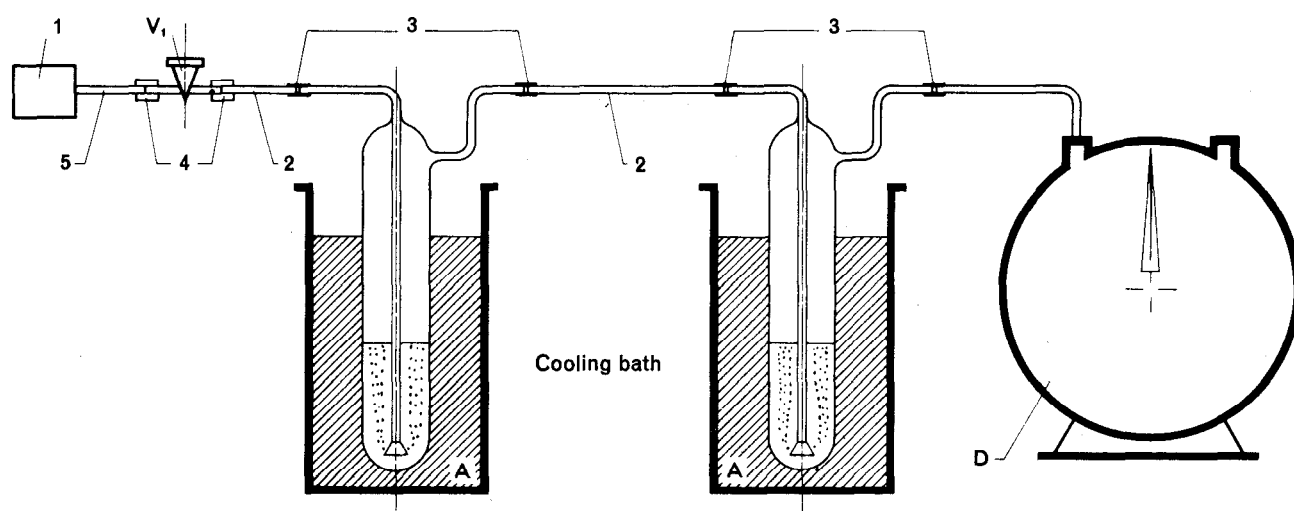
New sulphur hexafluoride shall not contain more than 10 ppm by mass of oil.



019 73.

(Dimensions in mm)

FIG. 6. — Gas wash bottle for absorption of oil from SF_6 gas.



020/73.

- A Absorption tube.
- D Gas-meter.
- V₁ Needle valve.
- 1 Brass fitting.
- 2 Glass tube: $\varnothing$ ext. 7 mm.
- 3 Union in silicone rubber or in fluorinated elastomer.
- 4 Metallic union.
- 5 Metallic tube: $\varnothing$ ext. 6-7 mm.

FIG. 7. — General design of the apparatus.

26. Dew-point method

Replace Under consideration by the following text:

26.1 Scope

This recommendation describes a simple apparatus and a test procedure with which the dew point of an insulating or other gas may be accurately determined. Using solid CO₂ as a coolant values of dew point may be measured down to approaching $-60\text{ }^{\circ}\text{C}$ with an accuracy of $\pm 1\text{ }^{\circ}\text{C}$.

26.2 Principle of the method

The condensation temperature of the impurities in a gas, that is the dew point of a gas, is measured by passing a sample of the gas through a closed cell in which is exposed a surface (almost always a metal mirror) the temperature of which is lowered artificially in a controlled manner. When the temperature of the surface is low enough, impurities in the gas, such as water or water plus other substances, condense on the surface forming a visible mist or "dew" or, at low temperatures, a crystalline "frost". That surface temperature at which this condensation remains stable, that is, neither grows nor diminishes with time, is for all practical purposes the dew point of the gas. However, it must be emphasized that, particularly at low dew points such as are normal for electrical insulating gases, the true dew point temperature at which condensation is stable is significantly higher than the temperature at which condensation is first observed — the actual difference depending on the instrument used and upon many other factors — and the temperature of first appearance cannot be used by itself to determine the dew point.

Notes 1. — The term dew point will be used for present purposes since this is the common usage in chemical and similar contexts irrespective of whether the deposit is actually a dew or frost. Different terminology is used in, for example, meteorological applications.

2. — As already pointed out, the measured dew point of an insulating gas is not necessarily an indication of the water content. However, in some circumstances it may be convenient to have available data of the pure water concentration corresponding to a given dew point. For this purpose, Appendix A shows values of saturation vapour pressure, absolute humidity, and ppm by mass of water in SF₆, for a pure water condensate. These data indicate the lowest concentration of pure water that can exist at a given dew point.

26.3 Dew-point instruments

Existing instruments for determining dew point fall into two broad categories:

a) Automatic instruments. Most of them are so constructed that the quantity of condensate is monitored by a photo cell, the output of which is used to control the mirror temperature so as to maintain a stable quantity of condensate; the mirror temperature should therefore be automatically held at the required dew point, which may be obtained by measuring the mirror temperature with a thermocouple, etc.

b) Manual instruments. These are made in numerous different designs, but have the common feature, that mirror cooling is controlled manually in response to the appearance or disappearance of condensate.

Note. — ISO/TC 125 is at present preparing a recommendation which will be a general survey of the requirements, different patterns, difficulties of use and operational precautions, etc., for condensation instruments.

Existing automatic instruments are generally expensive and often bulky; however, they can be used for present purposes, provided it is known that the mirror temperature is correctly indicated.

Existing manual instruments are difficult to use and erroneous readings are usually obtained at low dew points, which require the use of correction factors.

The instrument described in this recommendation has been developed to meet the need for an inexpensive instrument that can easily be used on site. It avoids most operational difficulties, is easily used by personnel with minimum experience and at the same time gives high accuracy.

Figure 8, page 16, shows the recommended dew-point cell in outline diagrammatic form. (Details of assembly, sealing, etc., which may vary from one pattern to another, are omitted.) The essential features of this design are as follows:

- a) Although an ordinary liquid-in-glass thermometer is used, the temperature of the cooled mirror surface is accurately measured by employing an appropriately designed thermometer pocket forming part of the isothermal mass on which the mirror is formed.
- b) The design ensures a high thermal resistance between the cooled mirror and the ambient air, so that leakage of heat to the mirror is small; this ensures that the mirror temperature is constant over its area and the same as the temperature of the thermometer pocket.
- c) Observation of first appearance of condensate is made very easy by two design features. Firstly, the cooled mirror, which is a disk about 1 cm in diameter made of polished nickel-plated copper, is surrounded without significant break of surface by a comparison mirror region which is always warmer than the cooled mirror and therefore remains free from condensation; secondly, a blackened screen is so arranged that all mirror surfaces appear black so long as no deposit is present. These two features ensure that when deposit appears it does so as a definite white disk on a black background, which is clearly and immediately observed even by those without previous experience of the instrument.

This instrument is intended to employ solid CO_2 in acetone as a coolant and, when used in accordance with the instructions below, gives accurate results better than $\pm 1^\circ\text{C}$ at dew points around -50°C , and more accurate results at higher dew points.

26.4 Apparatus

Dew-point cell

Essential features of the dew-point cell are shown in Figure 8; and described in Sub-clause 26.3.

Flowmeter

To indicate the flow of the tested gas in the range of 0.5 to 2 litres/minute approximately.

Note. — A float-type flowmeter is most suitable, but a *dry* gas meter and a watch or clock may be used instead. No great accuracy is required; indeed, the flowmeter can be dispensed with, but since some indication is needed that flow is taking place at an approximately constant rate and at about the suggested value, a suitable flowmeter is strongly recommended.

Solid carbon dioxide

A supply of solid CO_2 is needed. This may be purchased in blocks and broken up as required for use (the broken CO_2 may be kept for some days in a vacuum jar). Alternatively, devices are available for making solid CO_2 pellets from cylinders of CO_2 .

Temperature measurement

A liquid-in-glass thermometer of suitable range is usually the most convenient method of measuring the temperature in the thermometer well of the dew-point instrument. Thermometers should be available to enable temperatures between ambient and -60°C to be measured with an accuracy of at least $\pm 0.5^\circ\text{C}$. It is essential that these thermometers are calibrated or corrected for immersions of 3 cm (stem in ambient air) and that the lowest graduation is at least 14 cm from the base of the bulb; maximum diameter of bulb and stem: 7 mm.

Where more convenient, another temperature measuring device, which will measure temperatures in the present situation with the above accuracy, may be substituted.

26.5 Method

Clean the mirror surface of the cup and the inside and outside of the transparent casing with a soft duster. Assemble the cell, making sure the sealing rings or washers are clean and lubricated with a trace of silicone grease.

Figure 9. shows how it is recommended to connect the apparatus. Gas flows from the gas cylinder, through an all-metal fine control valve, if necessary, and through a sampling line as described in Clause 17.

From the sample line, the gas flows through the dew-point cell and finally through the flowmeter. The direction of flow through the cell is unimportant. Connect at least half a metre of 3 mm to 6 mm bore, stainless steel or polytetrafluoroethylene tubing between the cell outlet and atmosphere, without valves or other restrictions.

Notes 1. — The purpose of the length of outlet tubing is to prevent any contamination of the gas in the cell with water vapour from the outside atmosphere. This length of tubing may most conveniently be the tubing that connects the dew-point cell to the flowmeter.

2. — The test method described in this recommendation is designed to obtain the dew point at atmospheric pressure, and it is for this reason that there must be no restriction in the outlet line. A float-type flowmeter suitable for measuring gas flow does not constitute a significant restriction and will thus not significantly raise the pressure in the cell. For greatest accuracy, the atmospheric pressure should be noted; normal maximum variation of atmospheric pressure (about $\pm 10\%$) will change the observed dew point by roughly $\pm 1^\circ\text{C}$. Under unusual (e.g. high altitude) conditions the pressure shall always be measured and reported together with the test results.

3. — The cell itself may be suitable for tests at pressures above atmospheric; the manufacturer's data should be consulted. For tests at such higher pressures, a suitable pressure gauge and all-metal throttle valve should be connected to the outlet of the cell, ensuring that all other parts of the system are also suitable for the proposed pressures.

All tubing joints shall be air-tight and of all-metal (e.g. compression) type; alternatively, lengths of tubing may be butt-jointed with the joint covered by a tightly-fitting sleeve of polyethylene or polytetrafluoroethylene.

Put acetone into the inner cup of the dew-point cell to a depth of 3 cm to 5 cm (about 20 ml). Take care that no significant amount of acetone enters the space between inner and outer cup. Insert the thermometer into the thermometer well.

Note that in this cell there is a spot rather less than 1 cm diameter on the side of the cup adjacent to the thermometer well, which is the only point that is cooled; this is the only point at which a deposit will form. Position the cell in as good a light as possible, so that the cold spot is observed approximately tangentially with the eye approximately level with the cell. Then position the rotatable screen so that from the observation point the mirror surface reflects the blackened inside surface of the screen. Under these conditions, when a deposit forms the cold spot will appear white against a black background.

Establish flow of the gas to be tested through the cell. A flow rate of roughly 2 l/min will normally be found suitable at dew points of the order of -40°C or -50°C .

Notes 1. — Should the gas tested have a higher dew point, a lower flow rate may be adequate; e.g. at a dew point of 0°C , about 0.5 l/min is sufficient.

2. — Each test made in accordance with the procedure given in this recommendation will usually consume between 10 l and 40 l of gas (at atmospheric pressure), assuming that all pipework and equipment is dry prior to the test. Possible extensive drying out of the equipment may be avoided by keeping each item dry when not in use, e.g. by sealing with bungs, caps, etc., in accordance with normal low-humidity practice.

After gas has been flowing through the cell for at least 3 min to 5 min, drop small pieces of solid CO_2 into the inner cup, keeping watch on the thermometer and on the cold spot. The temperature indicated will fall steadily until each piece of solid CO_2 has evaporated; do not put a further piece of CO_2 into the cup until the previous piece has had its effect. Continue cooling until the cold spot appears to turn white, due to a deposit of condensate. Note the temperature at which this occurs, and discontinue adding further solid CO_2 . Also note the lowest temperature reached.

Allow the temperature to rise again and note the temperature at which the deposit finally disappears. The dew point of the gas in the cell is between this temperature and the temperature at which deposit first appeared.

This result should be regarded as a first trial run, and now that the required dew point has been roughly established, further readings should be made by re-cooling the cell. Cool the cell by not more than 2 °C at a time until a deposit is seen, then do not add further solid CO₂. For rough guidance, a piece of solid CO₂ about the size of a small pea will cool the cup by 1 °C to 2 °C. Do not stir the acetone.

Record temperatures t_1 , t_2 and t_3 in accordance with the notes below:

Temperature of first appearance, t_1

In the vast majority of cases, the first appearance takes the form of a white mist entirely and simultaneously covering the whole surface of the cold spot, so that the cold spot turns white quite suddenly. This occurs below the correct dew point: with the gas-flow rates quoted, it occurs about 0.5 °C to 1 °C below correct dew point for dew points between 0 °C and -20 °C, but the difference becomes progressively more important as the dew point falls even further (at -40 °C a deposit will not generally be first observed until the cup temperature is about 3 °C below correct dew point).

Lowest temperature reached, t_2

With the step-by-step cooling technique outlined above, where cooling is discontinued as soon as t_1 is observed, the cup temperature will not drop more than 0.5 °C to 1 °C below the temperature at which deposit is first observed. The temperature will then rise gradually. Particularly at dew points below about -20 °C, the deposit is likely to change from an overall apparent mist to a number of very small ice crystals; do not mistake this for the disappearance of the deposit.

Temperature of disappearance, t_3

Carefully note the temperature at which the deposit disappears entirely. If the above procedure has been correctly carried out, this will be not more than 6 °C to 8 °C above the lowest temperature reached (t_2).

26.6 Calculation of dew point

The dew point is calculated as the mean between the lowest temperature reached, t_2 , and the temperature of final complete disappearance of the deposit, t_3 .

Check that the temperature of the first appearance, t_1 , is an appropriate temperature below this mean value (in accordance with the above notes) and that the first visible decrease of deposit occurs at slightly above this calculated mean value.

Repeat the observations to check that a repeatable mean value is obtained (a repeatability of ± 1.0 °C is to be expected).

If a repeatable value is not obtained, purge the system thoroughly and make a new series of observations. If a repeatable value is still not obtained, and the system is made up of the recommended materials, a leak may be suspected.

26.7 Reporting of results

The report should include:

- a) the mean of valid measurements;
- b) the values of t_1 , t_2 and t_3 for each of the valid measurements;
- c) the atmospheric pressure.

APPENDIX A

HYGROMETRIC DATA FOR UNCONTAMINATED WATER VAPOUR

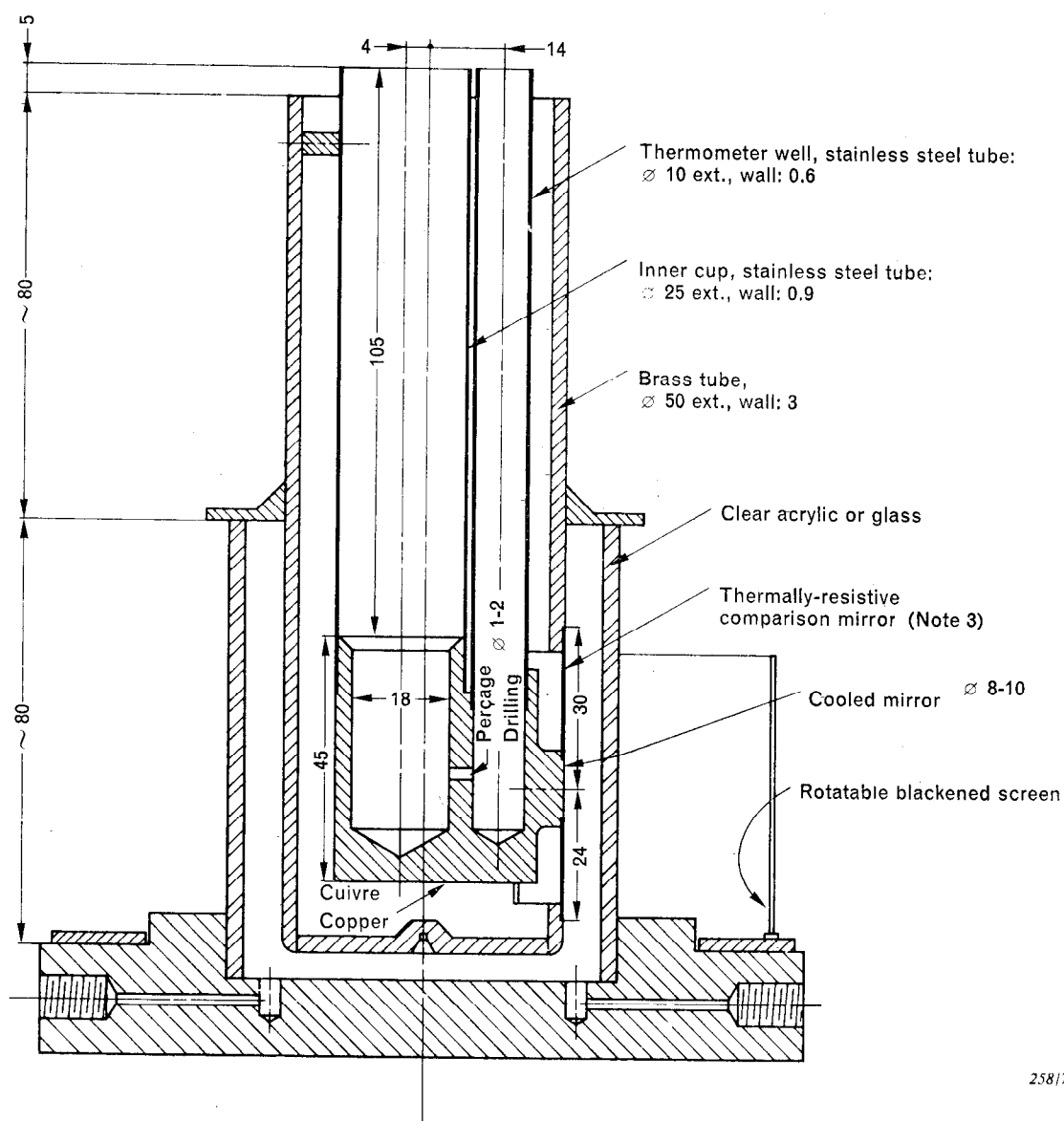
Dew point °C	SVP (see Note 1)		AH (see Note 2) g/m ³	ppm by mass in SF ₆ (see Note 3)
	torr	mbar		
20	17.527	23.36	17.30	2 840
19	16.471	21.95	16.26	2 670
18	15.471	20.62	15.26	2 510
17	14.524	19.36	14.33	2 360
16	13.629	18.17	13.45	2 210
15	12.782	17.04	12.61	2 070
14	11.982	15.97	11.82	1 940
13	11.226	14.96	11.08	1 820
12	10.512	14.01	10.37	1 700
11	9.889	13.11	9.71	1 600
10	9.204	12.27	9.09	1 490
9	8.606	11.47	8.49	1 400
8	8.041	10.72	7.93	1 300
7	7.510	10.01	7.41	1 220
6	7.010	9.34	6.91	1 140
5	6.540	8.72	6.45	1 060
4	6.097	8.13	6.01	989
3	5.682	7.57	5.60	922
2	5.291	7.05	5.22	858
1	4.925	6.56	4.86	799
0	4.581	6.11	4.52	743
-1	4.219	5.62	4.16	684
-2	3.880	5.17	3.83	629
-3	3.565	4.75	3.53	577
-4	3.280	4.37	3.25	532
-5	3.010	4.01	2.97	488
-6	2.765	3.68	2.73	448
-7	2.531	3.37	2.50	410
-8	2.322	3.09	2.29	376
-9	2.128	2.84	2.09	344
-10	1.945	2.59	1.92	316
-11	1.782	2.37	1.76	289
-12	1.630	2.17	1.61	264
-13	1.486	1.98	1.47	240
-14	1.359	1.81	1.34	221
-15	1.239	1.65	1.22	201
-16	1.130	1.51	1.11	183
-17	1.029	1.37	1.01	167
-18	0.936	1.25	0.922	152
-19	0.851	1.13	0.839	138
-20	0.774	1.03	0.763	125
-21	0.703	0.94	0.693	114
-22	0.638	0.85	0.629	103
-23	0.578	0.77	0.570	94
-24	0.524	0.70	0.516	85
-25	0.479	0.64	0.467	78
-26	0.429	0.57	0.423	70
-27	0.388	0.52	0.382	63
-28	0.350	0.47	0.345	57
-29	0.316	0.42	0.311	51

Dew point °C	SVP (see Note 1)		AH (see Note 2) g/m ³	ppm by mass in SF ₆ (see Note 3)
	torr	mbar		
-30	0.286	0.38	0.281	46
-31	0.256	0.34	0.253	41
-32	0.231	0.31	0.228	37
-33	0.208	0.28	0.205	34
-34	0.137	0.25	0.187	30
-35	0.168	0.22	0.165	27
-36	0.150	0.20	0.148	24
-37	0.134	0.18	0.133	22
-38	0.121	0.16	0.119	20
-39	0.108	0.14	0.106	17
-40	0.0962	0.128	0.094(8)	16
-41	0.0858	0.114	0.084(7)	14
-42	0.0766	0.102	0.075(4)	12
-43	0.0681	0.091	0.067(2)	11
-44	0.0607	0.081	0.059(8)	10
-45	0.0540	0.072	0.053(2)	8.7
-46	0.0479	0.064	0.047(2)	7.8
-47	0.0425	0.057	0.041(9)	6.9
-48	0.0377	0.050	0.037(1)	6.1
-49	0.0333	0.044	0.032(9)	5.4
-50	0.0295	0.039	0.029(1)	4.8
-51	0.0261	0.035	0.025(7)	4.2
-52	0.0222	0.030	0.022(7)	3.6
-53	0.0203	0.027	0.020(0)	3.3
-54	0.0178	0.024	0.017(6)	2.9
-55	0.0157	0.021	0.015(5)	2.5
-56	0.0138	0.018	0.013(6)	2.2
-57	0.0121	0.016	0.011(9)	2.0
-58	0.0106	0.014	0.010(4)	1.7
-59	0.0094	0.012	0.009(1)	1.5
-60	0.0081	0.011	0.008(0)	1.3

Notes 1. — SVP = Saturated Vapour Pressure. Values corresponding to dew points of 20 °C, 10 °C and 0 °C agree with the mean values in the International Skeleton Steam Tables (1963), intermediate values being in accordance with Bain's interpolation (National Engineering Laboratory Steam Tables, 1964). Values below 0 °C are from the Smithsonian Meteorological Tables, 6th Edition, 1951, and refer to conditions over ice.

2. — AH = Absolute Humidity. The value shown is the mass of water vapour contained in unit volume corresponding to the observed dew point of a sample taken from that volume. It is assumed a) that the sample is at the same pressure as that volume, and b) that the volume from which the sample was taken is at 20 °C.

3. — ppm = Parts per million. The water vapour content of SF₆, expressed in ppm by mass, is equal to $162.2 \times \text{SVP}$ where SVP is in torr, assuming the total pressure is 760 torr.



258/74

Notes 1. — All lower parts of the cup are nickel-plated and polished.
Main dimensions in millimetres.

2. — Base, cell, etc., shown in diagrammatic form; details of seals, assembly methods, etc., may vary and are therefore omitted.

3. — The thermally-resistive comparison mirror may be formed by letting in a thin plate of stainless steel, or by thinning the brass outer cup, or by any suitable method. The resistive zone extends over about 90° of the cup circumference.

FIG. 8. — Main features of dew-point cell design.

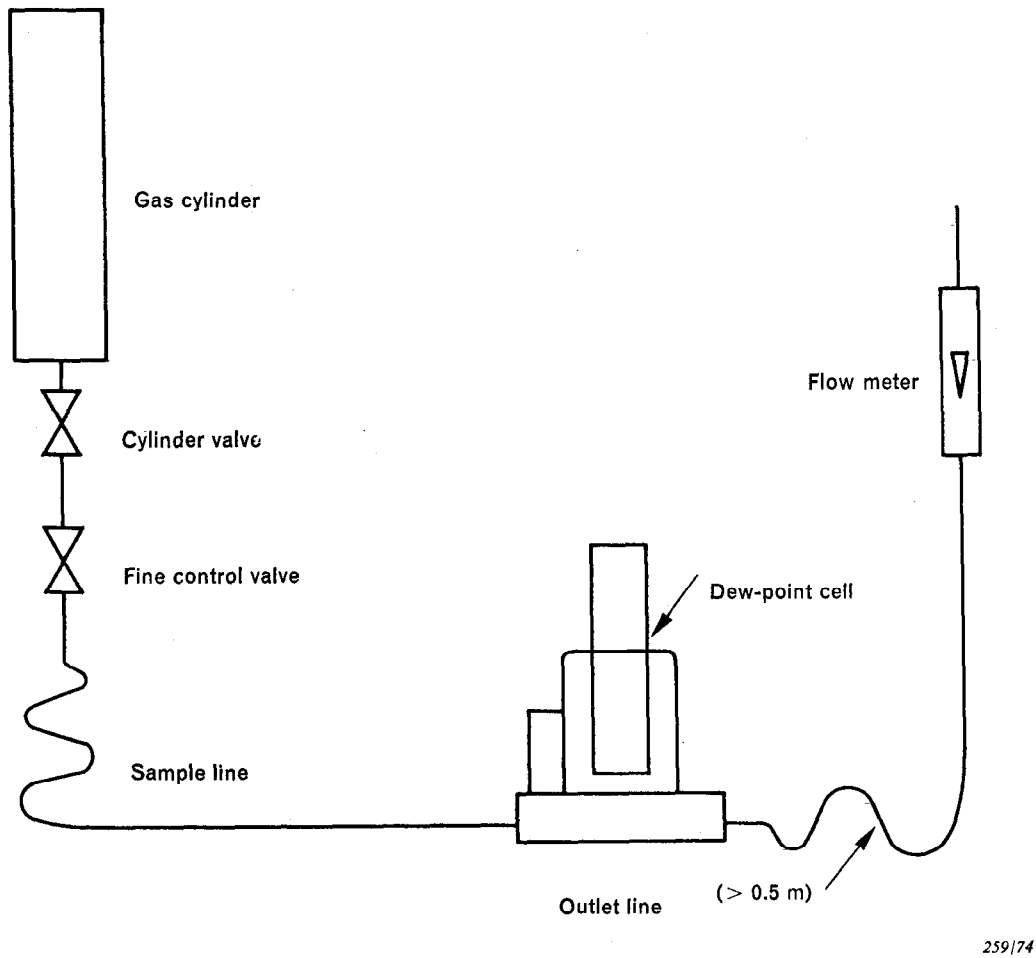


FIG. 9. — Arrangement for measurement of dew point.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standard Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type of grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Revision of Indian Standards

Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent in BIS giving the following reference :

Doc : No. ETD 03 (3246)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephone : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110001	{ 331 01 31 331 13 75
Eastern : I/14, C.I.T. Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
Southern : C.I.T. Campus, IV Cross Road, MADRAS 600113	41 29 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	6 32 92 95
Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. PATNA. THIRUVANANTHAPURAM.	